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A test method for deter	mining the co	ement content	of fresh con	ncrete was
developed for both labo	ratory and f	ield use. Us	ing hydrochle	oric acid
to keep a concrete mixt can be determined to wi	ure constant.	y neutralize	d, the cement	t content
Field tests indicate th	e method is n	nost useful i	n evaluating	the per-
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portions of a batch can	be determine	ed in about o	ne hour. Ad;	justments
to aggregate and/or cem mixing uniformity.	ent feed can	then be made	as needed to	o improve
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# STATE OF CALIFORNIA DEPARTMENT OF TRANSPORTATION DIVISION OF STRUCTURES & ENGINEERING SERVICES OFFICE OF TRANSPORTATION LABORATORY

September 1976

FHWA No. F-4-13(6) TL No. 635149C

Mr. C. E. Forbes Chief Engineer

Dear Sir:

I have approved and now submit for your information this final research project report titled:

#### CEMENT CONTENT OF FRESH CONCRETE

Study made by Roadbed & Concrete Branch
Under the Supervision of D. L. Spellman
Principal Investigator J. H. Woodstrom
Co-Investigator
Report Prepared by J. H. Woodstrom
Assisted by B. F. Neal

Very truly yours,

GEORGE W. HILL

Chief, Office of Transportation Laboratory

Attachment

BFN:1b

#### ACKNOWLEDGMENTS

The contents of this report reflect the views of the Transportation Laboratory which is responsible for the facts and the accuracy of the data presented herein. The contents do not necessarily reflect the official views or policies of the State of California, or the Federal Highway Administration. This report does not constitute a standard, specification, or regulation.

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# INTRODUCTION

The evaluation of adequately mixed concrete has been of concern to those involved in concrete work ever since concrete became a major structural element in a wide variety of facilities. In spite of vast technological changes in concrete plant design, mixing equipment and in improved specifications, the engineer cannot always safely assume that he has uniformly mixed concrete from one end of the mixer discharge to the other. The variations that occur, probably more often than we realize, are difficult to detect.

Mixer performance studies have proven to be valuable aids in determining the significant factors affecting concrete uniformity. Test programs are generally quite costly to run, require special sampling equipment, and take a considerable length of time for analysis because of the need for strength tests. There has been a notable deficiency in mixer performance tests because of the inability to determine the cement content of a sample of concrete in a short period of time under field conditions. Since a variation in cement content is the most significant factor affecting concrete quality, the need for its determination is of prime importance. Of considerably less significance are the more usual field measures used such as comparing slump or penetration, the amount of coarse aggregate per cubic foot of conctete, and unit weight. A large variation in cement content may have a relatively small effect on the tests mentioned above. Since other factors may also cause test variations, the conventional tests provide no early indication of cement imbalance. Thus, the development of a rapid test for determing cement content would provide the capability of providing adequate job control of freshly mixed concrete by testing for the most critical ingredient with respect to concrete quality.

#### CONCLUSIONS

Based on the laboratory and field tests reported here, the following conclusions are considered warranted.

- 1. The constant neutralization test is a fast and reasonably accurate method of determining cement content. Time required is 1 hour after beginning test. From the limited laboratory tests, accuracy appears to be within about 1/4 sack of cement per cubic yard.
- 2. A control must be established before determining cement content of project samples. A new control curve must be established if there is any change in cement or aggregate source.
- 3. At present, the most applicable use of the method is to test mixer efficiency. Test show that imbalances of any of the concrete ingredients during the mixer charging sequence can affect cement distribution within the batch. The test method can be used to determine within batch variations of cement distribution and thus provide information to aid in correcting the charging sequence.
- 4. The test was not proven to be of sufficient accuracy for routine control of cement content during normal concrete production.

#### IMPLEMENTATION

The test method developed during this research is not intended for use in a routine manner in the production of concrete. Personnel costs in conducting a test program would normally be considered high and specialized training is required. Sampling may require special equipment and delays in production. The uniformity of cement distribution is normally adequate.

Specifications could be written to include limits as to the variation in cement content as determined by the titration test. Invocation of the requirements would be at the discretion of the Engineer and would be triggered by any of several factors indicative of the possible production of nonuniform concrete. These would include, but not be limited to: (1) visual observations (2) within batch variations of slump, unit weight, or strength (3) nonuniform batching techniques (4) use of new batching or mixing equipment (5) indications of plant malfunction (6) historical knowledge of plant equipment.

# LABORATORY WORK

In an effort to find a rapid, simple test to determine the cement content of a sample of fresh concrete, initial efforts were directed at the possibility of using straightforward physical tests. These include the following:

# Wash-Out Method

This approach involves washing a sample of concrete through a 100 or 200 mesh sieve. Larger sieves are incorporated to prevent overloading and damage to the fine screens. The solid material passing the fine sieve will consist essentially of 100% of the cement plus the initial aggregate fines and any fines generated in the mixing process. Mixing water is also "lost" in this process. The total saturated surface dry weight of retained aggregate is subtracted from the initial weight of the concrete sample, the remainder being the total of the lost material; cement, aggregate fines and mixing water. If it can be assumed that the amounts of mixing water and aggregate fines are reasonably uniform throughout a batch of concrete, a fairly close indication of the uniformity of cement distribution can be obtained.

The washout approach has been used with reasonable success under certain conditions and an attempt was made to develop a rapid washing facility. An apparatus was constructed consisting of a large funnel in which a sample of fresh concrete was placed. Water under pressure was introduced from the bottom and the velocity and turbulence of the diluted concrete separated the fines from coarser aggregate particles. The fines were carried away through an overflow which was run across a 100 mesh sieve.

Both the equipment and the premise of the testing approach had weaknesses. The concrete samples in various portions of a

batch were found to be subject to imbalances which affected the indicated cement content. The test apparatus as designed was somewhat unwieldy and required constant attention to assure complete segregation of the fines. It was decided that it was unlikely that this approach could be developed to the point where adequate accuracy could be achieved for field testing.

#### Heavy Media Method

An effort was made to separate cement from mortar using a heavy liquid tetrabromoethane solution with vibration in an ultrasonic bath. Specific gravities of sand, tetrobromoethane and cement are approximately 2.65, 2.95 and 3.15, respectively.

Samples of wet concrete mortar mixed with tetrabromoethane were placed in polyethylene or glass centrifuge tubes. The tubes were then placed in water in the ultrasonic bath and vibration activated. Separation of sand and cement was unsatisfactory. Variations of the test procedure were tried, such as centrifuging the samples prior to placing in the ultrasonic bath, but none improved the test to a satisfactory degree. It was concluded that:

- 1. Cement is apparently too fine to be moved by gravity through the heavy liquid.
- 2. The attraction between cement and sand, when in a wet state, is too great for separation by ultrasonic vibration.

Attempts to develop this method further were abandoned.

# Constant Neutralization Method

Test Method No. Calif. 338, "Determination of Cement or Lime Content in Treated Aggregate by the Titration Method", has been used successfully in California for a number of years. This method is based on the theory that the concentration of hydroxide ions, liberated during the hydration process, is directly proportional to the amount of cement in an aggregate-cement mixture. By making a solution of the mixture, water and phenolphthalein indicator, hydrochloric acid can be constantly added to combine hydrogen ions with the hydroxide ions and keep the solution neutral. By measuring the amount of acid added over a standard period of time, the cement content can be determined.

The test method was developed basically for the purpose of checking the cement content of cement treated bases. These are lean mixtures, normally ranging from 3 - 5% cement by weight of the dry aggregate. Early trials with richer mixtures were disappointing because of rather large variations in the results. After considerable experimentation it was found that with certain modifications results could be obtained which were considered satisfactory. The primary change was to use only that portion of a mix passing the No. 4 sieve. Another change was to increase the amount of water added to a 150 gram sample from 400 to 1000 milliliters. The additional water makes color changes more definite and appeared to increase the accuracy of results. The test method as revised for use with concrete can be found in the appendix.

Many of the early tests were on concrete mixed by hand in small batches. A comparison between batches mixed by hand and those mixed by machine (pan type) indicated more consistent results were obtained from the machine mixes. Hand mixes were satisfactory provided certain precautions were observed, such as:

weighing materials accurately, stirring the mixture thoroughly, and taking care that no mortar was lost or left in the mixing pan. It was also found that for hand mixing, larger batches (20 pounds) gave better results than small batches (10 pounds). Tests on concrete mixed with a barrel mixer initially indicated less satisfactory results than obtained from the pan type machine mixes. Considerable improvement resulted from "buttering" the mixer or omitting washing of the drum between batches. Regardless of mixing method, the most important consideration in obtaining accurate results is the securing of a representative sample.

Laboratory tests were also made to determine the effect of varying cement or water with other ingredients held constant. In one case, three mixes were made in which aggregate weights and slump were held constant. The designed cement content was 6 sacks per cubic yard. One batch was mixed with cement added at that rate and in the other two, cement was varied plus and minus 10% from the designed factor. Table 1 shows the titration results for each mix.

TABLE 1
Batched Cement Content vs. Grams of Acid Used

Batched Cement Content, Sks./Cy.	Acid Added* Grams	Indicated Cement Content Sks./Cy.
5.4 (Designed -10%)	143.7	5.29
6.0 (Designed)	163.0	6.00
6.6 (Designed +10%)	177.7	6.54

<sup>\*</sup>Average of three tests, using 150 gram mortar sample.

The results indicate an accuracy within 2% or, in this case, about 1/8-sack/cy.

In another series of tests, aggregate and cement amounts were held constant and water varied to produce different slumps. Table 2 shows the results of these titration tests.

TABLE 2

Effect of Water-Cement Ratio on Titration Results

W/C Ratio	Slump, <u>Inches</u>	Acid Added,*Grams
0.32	1/4	247
0.34	3/4	245
0.36	1-1/4	240
0.38	2-1/4	235
0.41	4-3/4	237
0.43	5	236
0.45	8	229

<sup>\*</sup>Average of three tests using 150 gram mortar sample.

The results indicate that within the normal working range of 1 to 5 inches slump, variations due to water are not likely to be large. However, any control mix for a project should be made in the slump range that is expected to be used.

In the laboratory, it is common practice to check yield and cement content by the test method described in ASTM Designation: C138. Table 3 shows results of titration tests on concrete with various cement contents as determined from the yield test. Aggregate and cement sources and slump were held constant.

TABLE 3

Titration Results vs. Cement Content From Yield Test

	Content	Acid Added*	Indicated Cement Content Sks./Cu. Yd.**
Design	Yield Test		
5.0	5.20	185.7	5.20
	5.10	176.7	4.95
	5.07	179.7	5.03
5.5	5.58	198.3	5.55
	5.46	196.3	5.50
	5.53	195.0	5.46
6.0	5.99	210.3	5.89
	6.04	219.7	6.15
	6.18	214.0	5.99

<sup>\*</sup>Average of three tests.

These results also confirm that a good correlation exists between the amount of acid used for constant neutralization and the cement content of fresh concrete.

The test method was tried in the field on a few projects during concrete placements. The results indicated considerable variation in cement content between batches, but were inconclusive since results were not verified by other means. However, in 1971 an opportunity arose to make comprehensive testing possible in the field, some of which were under controlled conditions. The work is reported under the following section on implementation.

<sup>\*\*</sup>From first yield test - 185.7 grams = 5.20 sks.

#### FIELD IMPLEMENTATION

In 1971, a new concrete batching plant with twin 15-cubic yard mixers was used on a 17-mile paving project near Williams, California. At various locations serious defects were noted on the new pavement. Excessive cracking was evident and considerable raveling occurred when sawing weakened plane joints. Pavement cores taken in these areas showed the concrete to be deficient in cement. Wide variations in cement content were obvious within the confines of a single core in many cases. The problem was found to be related solely to nonuniform distribution of cement within a single batch of concrete.

When the source of the problem was determined, the contractor was notified that certain plant modifications were necessarry to assure proper batching. The contractor, in cooperation with the plant manufacturer, made these changes and on a subsequent paving project near Saugus, California, a special testing program was conducted to evaluate the mixing characteristics of the plant. The titration test proved to be a valuable tool in the test program that was conducted.

In the particular plant layout in question, the cement and water batching facilities are in a tower above the mixers. Cement is stored in an overhead silo, weighed, and then discharged directly into the mixing drum separately from the aggregate. Water is weighed and placed in an intermediate holding tank from which it is charged into the mixers. The aggregate is batched in a low profile portable weigh batcher, then a belt elevates it to the mouth of the mixing drums. Charging of the mixer is carried out in a pretimed sequence in order to blend aggregate, cement, and water. About 7 seconds is required for the aggregate to travel from the belt below the weigh batcher to the mixer

drums. At time zero, (arbitrary) the first aggregate gate opened, starting the sand feed. In sequence, the other two aggregate bin gates opened, layering these aggregates onto the sand. Cement and water are gravity fed from overhead directly into the mixer in timed sequence. The normal time of the operation in seconds is as follows:

	Time to Empty Hopper	Gate <u>Delay</u>	Time on Belt	Total Time for All Material to be in Drum
Sand	27	0	7	34
#2 Aggregate	25	1	7	33
#3 Aggregate	24	2	7	33
Cement	21	9	0	30
Water	(Not time	d)		

The gate on the cement hopper is activated by a step switch that opens it at a predetermined moment. When operating in the proper manner the step switch gate arrangement controls the rate of cement discharge so that it more or less corresponds in proper proportion to the time taken for the aggregate to be charged. At Williams there was some evidence that the cement hopper either opened early, before the aggregate reached the mixing drums, or it did not "step" properly, opening all the way, or nearly so, thereby putting most of the cement in as a slug which was not subsequently distributed inside the mixer. Corrective modifications made to the plant included installation of a visual and audible warning system which would indicate whether or not the cement was discharged at a controlled rate from the time discharge gate opened until no more than about 200 pounds of cement remained in the hopper. Criteria suggested for the rate of cement feed for normal operation was as follows:

Percent of Aggregate Charging Cycle Time	Percent of Cement to be in Mixer
25	15-35
. 50	40-60
75	65-85
110	100

In order to evaluate mixing characteristics, two series of tests were performed. Test Series One was conducted on December 21, 1971, and January 5 and 6, 1972, at Saugus. The purpose of the test program was to determine the effects of controlled imbalance of cement on the uniformity of concrete as well as to evaluate normal production. A tabulation of test data is shown on Tables 4 and 5.

Samples of concrete were obtained by stopping the mixer and physically entering it with sample containers and shovels. The "front" of the drum is that portion from where the concrete is discharged.

Details of plant operation and comments on batching procedures and test results for Test Series I follows:

February 21, 1971

#### Batch No. 1

North mixer intended to be normal operation. Step switch on the cement gate set to provide a charging period of 26 seconds, commencing 1-1/2 seconds after opening of first aggregate gate. The water valve was found to be set incorrectly, allowing a higher concentration of water near the rear of the mixer at the end of 50 seconds mixing. The wide dispersion of test results was somewhat alleviated by mixing an additional 20 seconds. (Batch 1a)

#### Batch No. 2

Intended to check North mixer under normal operation. Even though a slow delivery of the No. 2 rock was observed, test results indicate adequately mixed concrete. Additional 20 seconds of mixing had little effect on uniformity. (Batch 2a)

#### Batch No. 3

To check South mixer under normal operation. Adequate mixing apparent. Additional 20 seconds of mixing had little effect on uniformity. (Batch 3a)

#### Batch No. 4

To check normal operation of North mixer. Mixing adequate.

January 5 and 6, 1971

#### Batch No. 5

To check normal operation of South mixer. Cement feed found to require total of 37 seconds which may account for higher strength of concrete from rear of drum. Titration test did not differentiate cement concentration. Water and mortar spilled from front of mixer during mixing cycle.

#### Batch No. 6 - North Mixer

Cement concentrated near front of mixer as indicated by all test parameters. Additional mixing of 70 seconds did not provide concrete of satisfactory uniformity. (Batch 6a)

# Batch No. 7 - South Mixer

Similar results as in Batch No. 6.

#### Batch No. 8 - South Mixer

Cement charged in first part of batch, 300 seconds provided adequate mixing.

Batch No. 9 - North Mixer Similar to Batch No. 8.

#### Batch No. 10 - South Mixer

Slow feed of water (62 seconds) caused wide variation of test results between front and rear of mixer.

#### Batch No. 11 - South Mixer

Slow feed of water (62 seconds) again observed. Titration test indicates disparity in cement distribution, other tests indicate adequate uniformity. A sample can lid was found in the feed valve to the south mixer slowing flow of water.

#### Batch No. 12 - North Mixer

Normal operation and adequate uniformity.

# Batch No. 13 - South Mixer

Normal operation. Grout lost from front of mixer may have affected test results.

#### Batch No. 14 - North Mixer

Shortened cement feed and adequate mixing.

#### Batch No. 15 - South Mixer

Shortened cement feed (21 seconds) and adequate mixing.

# Batches Nos. 16 through 29

All batches sampled during production under normal operation. Adequate mixing in all batches except Batch No. 22.

# Production Testing, February and March 1976

Test Series 2 was conducted during actual paving operation and tests were performed by job personnel. Results are shown in Table 6 and indicate that adequately mixed concrete was produced.

TABLE 4
TEST SERIES I
SAUGUS TEST DATA SUMMARY
CONTROLLED CEMENT IMBALANCE

									Cement Content	•H	ve Strength	•
Bato & Lo	Batch Number & Location	Mixing Time, Secs.	Mixer	Pen., Ins.	Slump, Ins.	Air %	Unit Wt. Lbs./CF	Coarse Aggr. Lbs./CF	Sks/CY (Titra- tion)*	7-day Mortar Strength**	Cylind 7 days 2	iders* 28 days
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\*Average of 2 tests \*\*Averate of 3 2"x2" cubes

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TABLE

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TABLE 5 TEST SERIES I SAUGUS TEST DATA SUMMARY NORMAL STAGING OF CEMENT

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# DISCUSSION

In Test Series I, three sets of strength data were obtained because of the desirability of relating the effect of cement imbalance on strength. Variations in other properties, as determined by any of the tests on the plastic concrete may also have a significant effect on strength. The effects of the measured variations may be cumulative or offsetting from batch to batch making any statistical analysis relating cement content and strength invalid. To further complicate matters the source of coarse aggregate was changed several times with different combinations of the two primary sizes being used.

A plot of the cement content variation within a batch of concrete as determined by the titration test, and the associated differential in strength is shown in Figure I. It is seen that in spite of the variables affecting strength as mentioned above, a reasonably good relationship exists.

It has been well established that to obtain uniformly mixed concrete from central-mix plants, operating on short mixing time (50 seconds) proportionate blending of ingredients as they are charged into the mixer is essential. Under ideal batching conditions, acceptably uniform concrete can be obtained with as little as 30 seconds of mixing. With gross imbalances of any of the ingredients in the charging sequence, it is virtually impossible to correct the condition by any reasonable amount of extra mixing. The effects on concrete quality are most extreme when cement or water receive the charging imbalance.

Criteria for mixing efficiency as defined in the California Standard Specifications are:

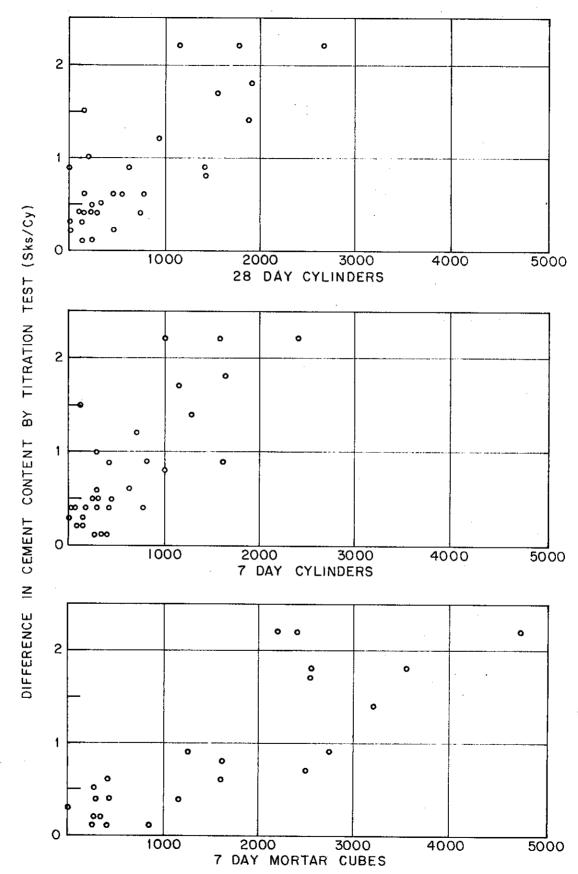


Figure I. EFFECT OF CEMENT VARIATION ON STRENGTH WITHIN BATCH

"The difference in penetration, determined by comparing penetration tests on two samples of mixed concrete from the same batch or truck mixer load, shall not exceed 1/2-inch. Variation in the proportion of coarse aggregates will be determined from the results of tests for 2 samples of mixed concrete from the same truck mixer load and the difference between the 2 results shall not exceed 6 pounds per cubic foot."

Other parameters can be used for concrete uniformity although no generally accepted limits have been established. An AASHTO-ARBA Joint Committee recommends the following:

Permissible Range Within Batch from 3 Samples (Not to be exceeded in 6 of 7 batches)

<u>Test</u>	Plant	Roadway
Slump, Inches	2.25	1.75
Air Content, %	2.0	1.5
Weight per Cu. Ft.	4.0	5.5
Coarse Aggregate per Cu. Ft., % 28-day Compressive Str.	11.0	9.0
% (Avg. of 2 cyls. per		•
Sample, as % of 3 sample average)	25	20

One reason distribution of cement has not been used as a measure of mixing efficiency is the lack of a suitable test method to determine the cement content of fresh concrete. Results cited above indicates that the titration test can fill this need. In fact, it appears that a good measure of cement distribution may be the most important parameter in evaluating mixer efficiency, and a better indicator of mix uniformity than the measures commonly used. (i.e.: weight per cubic foot, weight of coarse aggregate per cubic foot, slump or penetration, unit weight of air free mortar.)

#### SUMMARY

The titration test, using the constant neutralization method and under controlled conditions, is considered adequate to determine cement content of fresh concrete to within 1/4-sack per cubic yard. Field trials indicate the test is especially valuable in determining mixer efficiency. By checking different portions of a batch, aggregate and cement feed gates can be set and cycle times adjusted to provide proper mixing. Even after such adjustments, the results indicated between-and within-batch variations although these were not always verified by strength tests.

The cement content can be determined by the titration test in less than 2 hours after concrete is mixed. The test requires 1 hour after preparation of specimens. For control and routine field testing, three titration specimens are prepared and tested from each concrete sample. For mixer performance testing, only 2 specimens are prepared for each sample but since 3 locations in the mixer are sampled, there are 6 specimens to titrate at one time. This can readily be handled by one operator after brief practice. An experienced operator has been observed doing titrations on 9 containers at one time, but this is not recommended.

#### APPENDIX A

# DETERMINATION OF CEMENT CONTENT OF FRESH CONCRETE BY CONSTANT NEUTRALIZATION

#### SCOPE

This method describes a test for determining the cement content of fresh concrete. The determinations are based upon the constant neutralization by three normal hydrochloric acid of an aqueous solution of concrete mortar and water. This is accomplished by adding sufficient acid to just neutralize the hydroxide ions in the cement. When the aggregate is "neutral" or nonreactive, the amount of acid used is directly proportional to the cement content of the fresh concrete sample.

This method is divided into the following parts:

- I. Titration Procedure
- II. Determination of Calibration Curve
- III. Relative Variation of Cement Content Within a Batch
- Part I. Titration Procedure

#### PROCEDURE

#### A. Apparatus

- 1. 1 100 ml titrating burette or other device permitting slow, easily controlled addition of acid.
  - 2. 1 burette stand and burette clamp.

- 3. 1 5-gallon plastic "carboy" (to contain the 3N acid solution) equipped with siphon, neoprene or tygon tubing, hose clamps, etc., for connection to the titrating burette.
- 4. 9 2-quart wide mouth polyethylene containers with handles and flat bottoms.
  - 5. Graduated eye dropper or 10 ml graduated cylinder.
  - 6. 6 stainless steel stirring rods.
  - 7. No. 4 sieve, 12-inch diameter.
- 8. Balance or scale with 5 kg capacity, graduated to 1.0 gm.
  - 9. Glass or polyethylene bottle for burette overflow.
  - 10. 1 1000 ml graduated cylinder.
- 11. 6 250 ml Erlenmeyer flasks or polypropylene, three-way pour, disposable beakers.
- 12. Timer, graduated in 1 second intervals for a total of 1 hour.

#### B. Reagents

1. Hydrochloric Acid (approximately 3N). Pour the contents of two full standard 6-lb bottles of concentrated hydrochloric acid C.P. into the 5-gallon polyethelene carboy. Add tap water in 1-gallon increments until 5 gallons of solution is obtained. After each addition of water, shake the carboy vigorously for about one minute to obtain a homogeneous mixture. It is important to agitate this solution before starting each test series to maintain the homogeneous mixture.

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Note: When mixing the acid solution, always wear chemical protective gloves, goggles, and aprons. <u>DO NOT</u> use compressed air to agitate the solution in the carboy. <u>DO NOT</u> breathe acid fumes.

2. <u>Phenolphthalein Indication Solution</u> (1% solution). Dissolve 5 grams of phenolphthalein powder USP in 250 mls of ethanol. Dilute with 250 mls of distilled water.

#### C. Test Record Form

Record test data on "Field Laboratory Record of Constant Neutralization Tests", Form No. HMR T-597.

#### D. Test Procedure

- 1. Secure approximately 5 kg of a representative sample of fresh concrete and screen over a No. 4 sieve into a clean pan. The screen and pan should be dampened but excess water must be removed before screening. Screen as much of the mortar from the coarse aggregate as possible. Place the mortar in a covered container immediately after the screening operation. The screened mortar must remain covered and set no longer than 30 minutes before the addition of the 1000 mls of tap water. Testing should begin 45 minutes +15 minutes after mixing.
- 2. Prepare test specimens by weighing 150 gms  $\pm$  1 gm of the screened mortar into the 2-quart containers. Mix the screened mortar sample thoroughly before each sampling. Three specimens are required for each control and field sample and two specimens for each mixer performance test sample.
- 3. Add 1000 mls of tap water to each specimen and start timer.

- 4. Using the graduated eye-dropper or 10 ml graduated cylinder, add approximately 4 mls of phenolphthalein solution to each container. Stir for 5-10 seconds. (The solution will turn red due to the presence of cement.)
- 5. Place one stainless steel rod in each container, weigh, and record the weight of each container and its contents to the nearest 1.0 gm.
- 6. Fill each of the Erlenmeyer flasks with 60 mls of the 3N hydrochloric acid for initial acid. (The number of prepared flasks needs to equal the number of test specimens.) Acid may be measured out beforehand, but containers should be covered and not be allowed to set for more than one hour.
- 7. Ten minutes after the addition of water to the mortar, add 60 mls of acid to each 2-quart container of mortar solution and stir for 15 seconds.
- 8. At 1-minute intervals, following the addition of the 60 mls of acid, add approximately 5 mls of acid from the titrating burette and stir for 5 seconds. Follow this procedure dure for each of the containers in turn. Repeat this procedure until the color comes back slowly between additions. At this point the amount of acid being added at the 1-minute intervals should be reduced as necessary to just eliminate the color. (Excess acid may attack the aggregates in the later phases of the test, when the hydration products from the cement remaining in the mixture are low, resulting in an erroneous cement determination.) It is important that the operator exercise great care in maintaining an equally neutral solution in all specimens of a sample by diligently adding the acid necessary at the 1-minute intervals and stirring for 5 seconds.

- 9. After the 60 minutes have elapsed, reweigh, and record the weight for each container. From the difference between the initial and final gross weights, calculate and record the total weight of hydrochloric acid used to neutralize each of the three solutions (see Figure A).
- 10. The resulting weights of hydrochloric acid can be used to find an "average" cement content of the field test sample with the use of a calibration curve (see Part II), or to determine the variation in cement content for evaluation of mixer efficiency (Part III).

### E. Precautions

- 1. Use extreme caution when handling acid and use protective clothing and equipment. Do not breathe acid fumes.
- 2. This test cannot be performed by persons who are color blind.
- 3. Keep all reagent bottles closed when not in use to prevent evaporation and changes in concentration.
  - 4. Rinse and drain sample containers after every test.
- 5. The 2-quart containers should be inspected periodically for leaks.

# PART II. DETERMINATION OF CALIBRATION CURVE

# PROCEDURE

# A. Apparatus

- 1. Concrete Mixer. A revolving pan or revolving paddle mixer capable of thoroughly mixing batches of the prescribed size at the required slump.
- 2. Unit Weight Measure. A cylindrical watertight steel measure having a nominal capacity of 1/2 cubic foot, provided with handles. (See Test Method No. Calif. 518.)
- 3. Penetration or Slump Measure. (See Test Method No. Calif. 533 or ASTM Designation C143.)
- 4. US No. 4 sieve, brass, full height, 12 inches in diameter, for sieving fresh concrete. A rigid type extension and handles may be attached to facilitate handling and prevent spillage.

#### B. Test Procedure

- Central mix plant method optional.
- a. Prior arrangements with the contractor will be needed for mixing a batch 300 seconds, and it will be necessary to have an inspector to accurately record the scale weights for the 300-second batch.
- b. After the batch has mixed 300 seconds and discharged into a truck, take penetration measurements and secure samples at approximately one-third the distance from each end

of the truck keeping the two samples separate. The size of each sample should be sufficient to fill a 1/2-cubic foot unit weight bucket.

Note: If the penetrations are not within contract specifications, the batch should not be tested.

- c. After the completion of the unit weight tests on the two samples, proceed as described in Part I, D, of this test method. Precaution: Be sure samples are properly identified.
- d. Using the <u>scale</u> weights and an average of the two unit weights, compute the cement content for the batch in accordance with Test Method No. Calif. 518.

Note: Control batch should be mixed midway through the work day if only one control batch is mixed.

# 2. Laboratory Mix Method - Referee

- a. Secure a sufficient quantity of sand, aggregate, cement, and admixture for three batches of at least 1.0 cubic foot each, and batch in the proportions of the mix being investigated. Batching of the water and admixture, if used, should be done immediately prior to mixing.
- b. Mix the concrete in accordance with accepted laboratory procedures (Test Method No. Calif. 530, Section C).
- c. Measure the slump of the concrete immediately after mixing in accordance with ASTM Designation: C143. When removing material from the mixer, use care to obtain a representative sample.

Note: Lab test mixes are usually not of sufficient volume to permit determination of ball penetration.

- d. Measure the unit weight and calculate the cement factor in accordance with Test Method No. Calif. 518.
- e. Return the material used for slump and unit weight tests to the mixing pan and remix for 1 minute.
- f. Proceed as described in Part I, D, of this test method.
- C. Determination of a Calibration Curve
- 1. The calibration curve, for a particular mix design, establishes the relationship between the amount of hydrochloric acid used and the cement content.
- 2. Using the graph provided on Form HMR T-597, plot the average grams of acid used versus cement content as determined by Test Method No. Calif. 518. Draw a straight line from the origin of the chart to the plotted point.
- 3. To determine the cement content of a field sample, follow the procedure as described in Part I, D. Mark the average amount of acid used to titrate the sample on the ordinate of the chart, move horizontally until crossing the calibration curve, then vertically down and read the cement content on the abscissa.
- 4. In the event of any change in source of aggregate, cement, or working solution of acid, a new calibration curve must be established. Ordinarily, a new calibration curve should be established each day. On long projects, with no changes taking place, every other day should be sufficient.
  - 5. Each operator must establish his own calibration curve.

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PART III. DETERMINATION OF RELATIVE VARIATION OF CEMENT CONTENT WITHIN A BATCH.

### A. Test Procedure

- 1. Obtain samples as describe in Part II, B, 1, of this test method except that the scale weights are not necessary and the normal mixing time shall be used.
- 2. Perform the test procedure described in Part I, utilizing two specimens of mortar from each screened sample.
  - 3. Record results on Form HMR T-597.

#### B. Calculations

1. Determine the average number of grams of acid required to neutralize all specimens.

Note: If several batches are tested, the average should include all tests.

- 2. Plot on the graph, the average number of grams of acid versus the <u>design cement content</u> of the batch.
  - 3. Draw a line from the origin through the plotted point.
- 4. Plot the average grams of acid of each pair of duplicate specimens along the indicated line.
- 5. Read on the abscissa of graph, the indicated cement content for each sample to determine range (see Figure B).

CEMENT CONTENT, SACKS/ CU YD

STATE OF CALIFORNIA - DIVSION OF HIGHWAYS
MATERIALS AND RESEARCH DEPARTMENT

FIELD-LABORATORY RECORD OF

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MATERIALS AND RESEARCH DEPARTMENT STATE OF CALIFORNIA - DIVSION OF HIGHWAYS FIELD-LABORATORY RECORD OF

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s Jar Wt. Grams.	Stort Final 1403 1563 1399 1560			1400 1553	1393 1549	1552	1551	16 76.	3			
Gross Jar Wt. Grams.	Start	1403	1399		1400	1393	1400	1400	101	SACK	= 120	
Jar	N 0.	01	XX		E	3/	6	30		6.0	17.6120	
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Cement	Content																
Avg.	HCI Gms. Content	4/1	160.5		154.5			į	6/2/		1555		6		٨		
HC	Gms.	091	191		153			152	151		ERAGE		= 1555		706.25		
s Jar Wt. Grams.	Final	1563	1399 1560		1400 1553	1393 1549		1552	1521		TOTAL AVERAGE		200		585		
Gross Jar Wt. Grams.	Start	1403	6681		1400	5651		1400	1400		707		1.\		11		
Jar	No.	10	NN		3	3/		6	30				= 6.0 5ACK		OF VARIATION		(12)
Station		94			98			90	•				DESIGN =		RANGE OF VI		HMR T-597 (Orig 12-71)